PATENT SPECIFICATION

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DRAWINGS ATTACHED

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Inventor: FREDERIC CHANLER TUTTLE.

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COMPLETE SPECIFICATION

Preparation of Pelletized Solid Materials

We, THE DOW CHEMICAL COMPANY, a Corporation organised and existing under the Laws of the State of Delaware, United States of America, of Midland, County of Midland, State of Michigan, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention pertains to a process for the preparation of a material in a solid particle form. More particularly, it pertains to the preparation of a solid material from an aqueous solution.

There are many materials which may more conveniently be handled in solid form and are thus prepared from aqueous solutions. For example, alkali metal pentachlorophenates are finding considerable utility as biocides. The product heretofore produced generally had a tendency to disintegrate to form dust sized particles during handling and storage due to attrition and possibly to sublimation and recrystallization of the product. Since an alkali metal pentachlorophenate in dust form is an irritating chemical, this characteristic has hindered the acceptance of this chemical. When the chemical is used as a biocide, individuals are required to handle the product under varying conditions such that special equipment cannot be used to minimize or remove the irritating dust as would be possible in an industrial plant where the product is used only as a reactant. Thus, an alkali metal pentachlorophenate product in solid form which will not disintegrate readily on handling or in storage is greatly desired.

Spray drying and fluid bed evaporators are known. However, a satisfactory product has not been produced heretofore by these methods. By spray drying techniques, fragile, spherical shell type particles are obtained which very readily disintegrate. By the usual fluid bed evaporator techniques with these materials, a product of very fine particle size, an agglomerate of fine particles, or one of irregular particles having a rough surface with irregular projections is obtained. These products readily form dust size particles, especially pentachlorophenates.

In accordance with the present invention an improved process for continuously pelletizing water soluble solid materials by spray coating particles of the material with an aqueous solution thereof under drying conditions, has now been made available. This process substantially eliminates the dusting tendency of many solid materials and comprises spray coating particles of the material under evaporating conditions in one or more dilute zones within a fluidized bed of the particles, which zone(s) is formed in place by injecting steam into the bed in a direction transverse to the flow of the fluidizing air. The solution and steam are advantageously injected into the fluidized bed by passage through the orifices of a bifluid nozzle.

It is generally desirable to control the size of pellets produced by the process, partly in the interest of obtaining a more suitable product and partly to facilitate maintaining the fluidized state in the bed. It is, therefore, desirable to continuously remove pelletized material from the bed, to bring it into contact with screens to separate an undersize and an oversize material. The oversize material is then passed through a grinder and the ground oversize and the undersize materials returned to the fluidized bed at a point below the zone(s) of dilute fluidization. The process is particularly suited for use in making substantially spherical pellets of solid sodium pentachlorophenate the diameters of which are in the range from 0.149 to 4.76 mm by spray coating particles of sodium pentachlorophenate with a water solution of sodium pentachlorophenate.

The invention may be more clearly under-

stood when considered in conjunction with the accompanying drawing which schematically illustrates an apparatus in which the process of the invention may be carried out.

As shown in the drawing a vessel 1 having a confined zone is assembled with a cyclone 2, an elevator 3, screen assembly 4, a grinder 5, and a heater 6. As shown in the drawing vessel 1 is a cylindrical vessel having a screen or perforated plate 10 located near the bottom. Below the screen or perforated plate an air inlet 11 is provided which is attached to one end of heater 6 by line 12. By means of a blower 13 and line 14 air can be blown through heater 6 and into the bottom of vessel 1 through line 12. At the top of the vessel a gas outlet is provided which is connected by means of line 16 to a cyclone 2 or some other dust collecting apparatus. A product outlet 17 is provided near the bottom of vessel 1 above the screen through which the product by means of line 18 may be discharged to elevator 3. The elevator discharges into screen assembly 4 where size separations can be made. The screen assembly, as shown, is equipped with three outlets, an outlet for oversize particles connected to grinder 5 by line 19, a product outlet attached to line 21, and an outlet for undersize particles attached to line 22.

Vessel 1 is provided with an inlet line 24 which extends into the vessel at an angle from the horizontal plane to discharge downwardly above the screen or perforated plate 10 near the center of the vessel. Means for air flow within line 24 is provided. The fine solid particles recovered in the cyclone 2 are discharged in line 24 as well as the fine particles from screen assembly 4 and the discharge from grinder 5 through line 26. Air locks 27, 28 and 29, respectively, are provided in lines 22 and 26 and the line from the cyclone to keep the air in line 24 from discharging into the respective equipment.

As shown in the drawing a bifluid nozzle 31 is positioned to discharge into the vessel along a horizontal plane. A number of such nozzles may be used, spaced apart around the periphery of vessel 1, however, in the drawing only one is shown.

In the operation of the apparatus, particles of solid material smaller than that desired are charged into vessel 1 through line 24. By means of blower 13 air is blown through heater 6 to be heated and then discharged into vessel 1 beneath screen or perforated plate 10 at a rate such that the particles added into vessel 1 will become fluidized. Steam at superatmospheric pressure, being injected through nozzle 31, atomizes the solution being added to the nozzle and creates a zone of dilute fluidization in the vicinity of the nozzle outlet. Particles in the fluidized bed passing through the dilute fluidization zone become coated wtih the solution in the portion of the atmosphere having a high steam content.

A portion of the particulate solid material is continually withdrawn from the fluidized bed through pipe 18 and elevated by means of elevator 3 to screen 4 where the product is separated according to size. Particles which are larger than the desired size are discharged through line 19 to a grinder 5 where they are ground and returned to the bed through line 24. The desired product taken off is discharged through line 21 and placed in storage or packaged as desired. The undersize particles leaving the bottom of the screen are discharged through line 22 into line 24 to be returned to the fluidized bed. The air used in drying and fluidizing the bed is discharged from vessel 1 through pipe 16 through a dust collecting apparatus such as cyclone 2 as shown. The fine particles recovered in the cyclone 2 are likewise discharged into line 24 and returned to the bed. The fine particles recovered by the dust collection equipment, the undersized product from the screen assembly, and the grinder output, are injected into the bed through line 24 by use of a carrying fluid such as air. These fines are injected into the bed near the center of the bed and below the zone(s) of dilute fluidization.

To obtain particles of the desired size it is essential that the solution be injected in the dilute fluidization zone created by the steam. The theoretical explanation for this is not definitely known but it can be speculated that in coating the particles in an atmosphere containing a high proportion of steam the particle surface is conditioned so that a good adhesion is obtained upon drying or evaporating the water from the solution coating the particles. While the solution and the steam may be injected separately, it is most convenient generally to use a bifluid nozzle and utilize a portion of the steam pressure to atomize the solution.

The term "dilute fluidization" is used herein under its accepted meaning to define dilute phase fluidization wherein the voids between the particles are very large and the particles are entrained in the fluid medium. This phase is usually separated from the dense phase fluidization or the normal fluidization phase by a slugging phase.

The dilute fluidization zones created by the steam must be entirely within the fluidized bed or entirely within the dense fluidization phase of the bed. The zone is thus encompassed by particles in a dense fluidization phase. The steam is injected into the bed in a direction transverse to the flow of the fluidizing air. It may be very conveniently obtained by injecting steam from the outer periphery of the bed. A single zone or a multiplicity of zones may thus be created by injecting steam from numerous points

from around the outer periphery of the bed. When a multiplicity of such zones are used within the bed, it is essential that the dilute fluidization zones do not intersect or overlap. Generally the zones created do not extend beyond 1/3 the distance across the cross-sectional area of the bed.

As pointed out above the zones of dilute fluidization and the introduction of solution may be obtained simultaneously by use of a bifluid nozzle. For example, in the preparation of sodium pentachlorophenate having a particle size in the range of 4 to 100 mesh (diameters of 4.76 to 0.149 mm), a bifluid nozzle may be used using steam at pressures in the range of 35 to 85 pounds per square inch (3.5 to 6.5 atm.). A ratio of steam to solution is generally maintained in the range of 0.05 to 0.60, preferably of 0.1 to 0.25 parts by wt of steam per part by wt of solution. Generally at the lower rates of solution injection lower steam pressures may be used. In operating it is generally preferred to have a zone of dilute phase fluidization within the bed which extends from about 12 to 24 inches (30 to 60 cm) within a bed having a diameter of 183 cm or more. A divergent nozzle is generally used so that the zone created within the bed has a diameter which is from about 1/8 to 1/3 the length of the zone. When the dilute fluidization zones are too large, a sufficient number of particles does not pass through the zone resulting in an increase in the proportion of undersized particles. Generally, the zones of dilute phase fluidization are maintained such that the total influence of the zones or the dilute phase fluidization is in the range of from 5 to 12 percent of the total volume of the fluidization bed.

The solution injected into the zone of dilute fluidization is generally relatively saturated so that less water would have to be vaporized. The air used for fluidization is preheated to a temperature to maintain the bed below the décomposition temperature of the product being handled and the amount of air is adjusted to give the desired fluidization. It is apparent that the amount of solution introduced must be such that the air being used is sufficient to dry the product.

The fine particles of the desired material being introduced into the bed to be built up must be introduced at a point below the dilute fluidization zones. Generally a carrying fluid is desirable so that the fine particles are immediately distributed and not segregated within the bed. It is preferred to inject the small size particles near the bottom of the fluidized bed or just above the screen transversely to the fluidized air flow and downwardly. The discharge should be positioned so that it does not discharge into zones of dilute fluidization.

The above process is especially applicable to the preparation of an alkali metal pentachlorophenate. The product produced resists attrition and does not sublime and recrystallize to form dust particles upon storage. However, it is apparent that the process may be used for the preparation of other materials from aqueous solutions. Illustrative examples of other materials which may be so prepared are the alkali and alkaline earth matel chlorides as sodium or calcium chloride, alkali and alkaline earth metal silicates, and inorganic salts of organic materials such as the alkali metal sulfonates of alkylated diphenyl oxide or other detergents. The products obtained are generally in a high density, spherical shaped particle form.

EXAMPLE

An apparatus similar to that shown in the drawing was assembled and sodium pentachlorophenate having an 18 to 40 Tyler mesh particle size (diameters of 1.00 to 0.42 mm) was prepared. The fluidized bed was 8 feet (244 cm) in diameter and fluidizing air heated to about 220°C. was used. The superficial velocity within the bed was varied in the range of 2.7 to 3.1 feet (82 cm to 94.5 cm) per second. Numerous runs were made where the height of the bed was varied from around 12 to 30 inches (30 to 76 cm). Around the outer periphery of the bed, six bifluid nozzles were uniformly positioned so that the distance of the nozzle above the grid or screen was from 0.2 to 0.4 times the height of the bed. The nozzles were positioned to discharge directly into the bed. The undersized particles were injected into the bed by use of air at a point near the center and just above the screen.

The portion continuously withdrawn from the bed had the following analysis:

Tyler Screen Mesh	Maximum Opening mm	Weight Percent on Screen	Accumulative Percent
14	1.40	12	12
18	1.00	16	28
25	0.71	19	47
30	0.59	16	63
35	0.50	20	83
40	0.42	8	91
45	0.35	4	95
50	0.297	3	98
70	0.210	1	99
100	0.149	1	100
thru 100		trace	

After screening a product about 63 percent of the fraction was obtained which had a size range between 18 and 40 Tyler mesh (diameters of 1.00 to 0.149 mm).

The particles which passed through the 40 mesh screen were returned to the bed, while the particles which were retained on the 18 mesh screen and larger were passed through the grinder. The product from the grinder had the following screen analysis:

Tyler Screen Mesh	Maximum Opening, mm	Weight Percent on Screen	Accumulative Percent
14	1.40	6	6
18	1.00	11	17
25	0.71	14	31
30	0.59	12	43
35	0.50	18	61
40	0.42	11	72
45	0.35	6	78
50	0.297	6	84
70	0.210	7	91
100	0.149	4	95
thru 100		5	100

The fines from the cyclone amount to approximately 3 percent by weight of the portion being withdrawn continually from the bed. Thus on the basis of 100 parts by weight of material being continually withdrawn from the bed, 63 parts of product, 28 parts of oversize, and 9 parts of undersize were obtained. The 9 parts of undersize particles, the 28 parts of oversize after grinding and about 3 parts of fines from the cyclone were returned to the bed to be spray coated.

In the operation a steam pressure of 40 to 45 pounds per square inch (4 to 4.4 atm) was used to inject an approximately 32 percent solution of sodium pentachlorophenate at a rate of about 10 pounds per hour per square foot (4.9 gm/cm^2) of the fluidized bed. At this steam pressure it was estimated that the zones of dilute fluidization obtained at each of the nozzles was approximately 15 inches (38 cm) in length and 5 inches (12.7 cm) in diameter. The bed temperature was about 110°C.

In the manner described above the run was repeated except that the steam pressure was varied from 35 to 50 pounds per square inch (3.5 to 4.5 atm) and a satisfactory product was still obtained. Above 50 pounds (4.5 atm) dusting was obtained. When the rate of solution injected, however, was increased to 20 pounds per hour per square foot (9.8 gm/cm^2), the optimum steam pressure to obtain the desired dilute zone was in the range of 55 to 65 pounds per square inch (4.7 to 5.2 atm). Pressures up to 85 pounds (6.8 atm) produced a satisfactory product without excessive dusting.

The location and the height of the nozzle above the screen in the bed was varied and it was found that a satisfactory product could be produced as long as the distance above the screen was such that the dilute zone of fluidization did not contact the screen or extend outside of the bed at the top.

In a manner similar to that described above, calcium chloride and sodium sulfonate of alkylated diphenyl oxide were prepared in a spherical shaped, high density particle form.

WHAT WE CLAIM IS:—

1. A continuous process for pelletizing a water soluble solid material which comprises spray coating particles of the material with an aqueous solution thereof under evaporating conditions in one or more dilute zones within a fluidized bed of the particles which zone(s) is formed in place by injecting steam into the bed in a direction transverse to the flow of the fluidizing air.

2. A process as claimed in claim 1 wherein the solution and the steam are injected into the fluidized bed by passage through the orifices of a bifluid nozzle.

3. A process as claimed in claim 1 or claim 2 wherein pelletized material continuously removed from the bed is brought into contact with screens to separate undersize and oversize materials, the oversize material is passed through a grinder and the ground oversize and undersize materials returned to the fluidized bed at a point below the dilute zone(s).

4. A process as claimed in any one of claims 1 to 3 wherein the water-soluble solid material is fine solid particles of sodium pentachlorophenate which are spray coated with an aqueous solution of sodium pentachlorophenate to produce substantially spherical pellets of solid sodium pentachlorophenate the diameters of which are within the range of 0.149 to 4.76 mm.

5. A process for pelletizing water soluble solid material substantially as described in the specific Example.

6. A process for pelletizing water soluble solid material substantially as described and illustrated with reference to the accompanying drawing.

7. Pelletized materials whenever obtained by the process claimed in any one of claims 1 to 6.

BOULT, WADE & TENNANT,
111 & 112 Hatton Garden,
London, E.C.1.
Chartered Patent Agents,
Agents for the Applicant(s).

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1,026,329 COMPLETE SPECIFICATION

1 SHEET

This drawing is a reproduction of the Original on a reduced scale.